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रंग रोगन के लिए बेरियम सल्फेट पिगमेंट —  
विशिष्टि  
( दूसरा पुनरीक्षण )

Barium Sulphate Pigments for  
Paints — Specification  
( Second Revision )

ICS 87.060.10

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## FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Raw Materials for Paints, Varnishes and Related Product Sectional Committee had been approved by the Chemical Division Council.

The first revision of the standard was done in 1972, in order to address the technological advancements registered in the manufacturing of pigments. In the first revision, new requirements such as *pH* of aqueous extract, relative density had been incorporated. The first revision also superseded IS 65 : 1950.

Now, this revision has been taken to bring out the standard in the latest style and format of the Indian Standard. It also incorporates amendments issued to the last version of the standard. In addition, the following changes have been made:

- a) Grades classification of type 1 material has been changed on the basis of barium content rather than material size;
- b) Carbonates content changed from 2.24 percent to 2 percent for both grades of type 1 and 0.45 percent to 0.5 percent for type 2;
- c) Residue on sieve has changed from 0.1 percent to 0.25 percent by mass for type 2; and
- d) Requirement for particle shape has been deleted.

The composition of the Committee responsible for formulation of this standard is given in Annex C.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard

# Indian Standard

## BARIUM SULPHATE PIGMENTS FOR PAINTS — SPECIFICATION

( Second Revision )

### 1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for barytes (naturally occurring barium sulphate) and blanc fixe (the precipitated barium sulphate). The material is intended for use as extenders for paints.

### 2 REFERENCES

The standards given below contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this Indian Standard are encouraged to investigate the possibility of applying the most recent editions of these standards:

<i>IS No</i>	<i>Title</i>
IS 33 : 1992	Inorganic pigments and extenders for paints — Methods of sampling and test ( <i>third revision</i> )
IS 101 (Part 8/ Sec 5) : 2022	Methods of sampling and test for paints varnishes and related products: Part 8 Tests for pigments and other solids, Section 5 Lead restriction test ( <i>fourth revision</i> )
IS 265 : 2021	Hydrochloric acid — Specification ( <i>fifth revision</i> )
IS 266 : 1993	Sulphuric acid — Specification ( <i>third revision</i> )
IS 1070 : 2023	Reagent grade water — Specification ( <i>fourth revision</i> )
IS 1303 : 1983	Glossary of terms relating to paints ( <i>second revision</i> )
IS 4825 : 1982	Specification for liquid-in-glass solid-stem reference thermometers ( <i>first revision</i> )

### 3 TERMINOLOGY

For the purpose of this standard, definitions given in IS 33 and IS 1303 shall apply.

### 4 TYPES

The material shall be of the following two types.

**4.1** Type 1 material shall consist of naturally occurring barium sulphate which may be bleached and shall have two grades:

- a) Grade 1; and
- b) Grade 2.

**4.2** Type 2 material shall be the precipitated barium sulphate.

### 5 REQUIREMENTS

#### 5.1 Form and Condition

The material shall be in the form of dry powder or in such a condition that it can be reduced to the powder form by crushing, without grinding action, under a palette knife.

#### 5.2 Lead Restriction

The material shall be tested for restriction from lead in accordance with IS 101 (Part 8/Sec 5). When thus tested the material shall not contain lead or compounds of lead or mixtures of both, calculated as metallic lead exceeding 90 ppm (*see* NOTE).

NOTE — When no lead is used during production, the product is considered lead free.

#### 5.3 Toxic Heavy Metal Restriction

Product shall not be manufactured using mercury and mercury compounds, cadmium, chromium VI, arsenic, antimony, and their oxides. The material shall not contain more than 0.1 percent by weight in total of above toxic heavy metals in the form of natural impurities or impurities entailed by the production process which are contained in the raw material when tested by the relevant atomic absorption spectroscopic methods (*see* NOTE).

NOTE — The test method standard for toxic heavy metal restriction is under development.

**5.4** The material shall also comply with the requirements given in Table 1.

### 6 PACKING AND MARKING

#### 6.1 Packing

The material shall be suitably packed as agreed to between the purchaser and the supplier.

#### 6.2 Marking

**6.2.1** The containers shall be marked with following details:

- a) Name of the material;
- b) Manufacturer's name and trade-mark, if any;

- c) Mass of the material;
- d) Lead content (maximum);
- e) Toxic heavy metals content;
- f) Batch number or lot number in code or otherwise;
- g) Month and year of manufacture; and
- h) A cautionary note as below:
  - 1) Keep out of reach of children, or
  - 2) This product may be harmful if swallowed or inhaled.

**6.2.2** The containers may also be marked with the Standard Mark.

### 6.2.3 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act, 2016* and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

### 7 SAMPLING

Representative samples of the materials shall be drawn as prescribed in IS 33.

**Table 1 Requirements for Barium Sulphate Pigments**

(Clauses 5.4 and 8.1)

SI No.	Characteristic	Requirement			Method of Test, Ref to	
		Type 1		Type 2	Annex	Clause No. in IS 33
		Grade 1	Grade 2			
(1)	(2)	(3)	(4)	(5)	(6)	(7)
i)	Compositions				<b>A-1</b>	—
	a) Barium (as BaSO4), percent by mass, <i>Min</i>	95	90	97		
	b) Carbonates (as BaCO3), percent by mass, <i>Max</i>	2	2	0.5	<b>A-2</b>	—
ii)	Volatile matter, percent by mass, <i>Max</i>	0.5	0.5	0.5	—	<b>6</b>
iii)	Residue on sieve, percent by mass, <i>Max</i>	0.25 on 40 micron IS Sieve (400 mesh)	0.25 on 60 micron IS Sieve (240 mesh)	0.25 on 40 micron IS Sieve (400 mesh)	—	<b>7</b>
iv)	Oil absorption ( <i>see</i> NOTE)	6 to 12		15 to 30	—	<b>8</b>
v)	Colour	A close match to that sample approved			—	<b>9</b>
vi)	Matter soluble in water, percent by mass, <i>Max</i>	0.5			—	<b>12</b>
vii)	<i>pH</i> of the aqueous extract	6 to 10			—	<b>14</b>
viii)	Relative density at 25/25 °C, <i>Min</i>	4.3			B	—
NOTE — This shall, however, be within + 10 percent of the approved sample, if any.						

## 8 TEST METHODS

**8.1** Tests shall be conducted as prescribed in relevant clauses of IS 33 and in Annex A to Annex C. References to annex and IS 33 are given in col (6) and col (7) of Table 1 respectively.

### 8.2 Quality of Reagents

Unless specified otherwise, pure chemicals and

distilled water (*see* IS 1070) shall be used.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

## 9 CRITERIA FOR CONFORMITY

A lot shall be declared as conforming to the requirements of this standard if the test results of composite sample satisfy the requirements prescribed under **5**.

## ANNEX A

[Table 1, Sl No. (i)]

## DETERMINATION OF BARIUM SULPHATE AND CARBONATES IN BARIUM SULPHATE PIGMENTS

## A-1 DETERMINATION OF BARIUM SULPHATE

## A-1.1 Outline of the Method

The material is fused with fusion mixture and the fused mass disintegrated with water. The barium carbonate formed is filtered off, washed free of any sulphate, dissolved in dilute hydrochloric acid and from this solution total barium, precipitated as sulphate, is weighed. Barium sulphate content is calculated after correcting the mass for the sulphate formed from the barium carbonate present in the pigment as determined in A-2.3.

## A-1.2 Apparatus

A-1.2.1 Conical Flask — 250 ml

A-1.2.2 Oven — capable of maintaining temperature of  $105\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ A-1.2.3 Muffle Furnace — capable of maintaining temperature up to  $600\text{ }^{\circ}\text{C}$ 

A-1.2.4 Asbestos Pad Gooch Crucible

A-1.2.5 Desiccator

A-1.2.6 Platinum Crucible

## A-1.3 Reagents

A-1.3.1 Hydrochloric Acid — approximately 1 N (see IS 265)

A-1.3.2 Dilute Sulphuric Acid — approximately 6 N (see IS 266)

A-1.3.3 Anhydrous Sodium and Potassium Carbonates (Fusion Mixture) — 1 : 1 by mass

A-1.3.4 Dilute Hydrochloric Acid — 1 : 1 by volume

A-1.3.5 Ammonium Hydroxide Solution — of relative density 0.88

A-1.3.6 Sodium Carbonate Solution — 3 percent (m/v)

A-1.3.7 Ammonium Sulphate Solution — 10 percent (m/v)

A-1.3.8 Methyl Red Indicator — 0.03 percent solution in water (m/v)

## A-1.4 Procedure

## A-1.4.1 Determination of Barium Sulphate

Weigh accurately about 0.5 g of the sample and mix

with about 10 times its mass of fusion mixture using an agate mortar if necessary. Fuse the mixture in a platinum crucible for about an hour. Transfer the crucible, when partially cooled, to a 250 ml beaker containing about 100 ml hot water. Heat the beaker to disintegrate the melt and to get it completely dislodged from the crucible. Remove the crucible, after carefully washing it with a jet of water to free it from any adhering residue. Filter the contents of the beaker through a Whatman No. 2 or equivalent filter paper and wash the residue with the 3 percent hot sodium carbonate solution, till the washings give no positive test for sulphates. Transfer the residue to the (now empty) 250 ml beaker, by piercing the filter paper and washing the residue down with a jet of water. Wash down the filter paper with few ml of hot dilute hydrochloric acid and finally with hot water into the beaker. The residue will have been completely dissolved. Add few drops of methyl red indicator, neutralize excess acid by ammonia solution added dropwise, and make acidic again with few drops of dilute hydrochloric acid. Add water if necessary to make up the volume to about 150 ml and boil. Gradually add slight excess of the 10 percent ammonium sulphate solution and continue gentle boiling for 10 min to 15 min more to render the precipitate granular and easily filterable. Stand the precipitate for some time and filter through a previously ignited and tared asbestos gooch crucible. Wash with hot water, heat in an oven for 15 min to 20 min at  $105\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ , ignite at about  $600\text{ }^{\circ}\text{C}$  in a muffle furnace for 30 min to 40 min, cool and weigh.

## A-1.5 Calculation

Barium sulphate, percent by mass

$$= \frac{100M_1}{M_2} - 1.182$$

where

$M_1$  = mass, in g, of the residue in the gooch crucible;

$M_2$  = mass, in g, of the material taken for test; and

$B$  = barium carbonate percent by mass as determined in A-2.3.

## A-2 DETERMINATION OF ACID SOLUBLE SALTS EXPRESSED AS BARIUM CARBONATE FOR DETERMINATION OF BARIUM CARBONATE

### A-2.1 Outline of the Method

Barium carbonate from a known quantity of material is extracted with dilute hydrochloric acid, precipitated as barium sulphate and barium carbonate calculated from the mass of the sulphate.

### A-2.2 Procedure

Weigh accurately about 5 g of the material and transfer to a 250 ml conical flask. Add about 60 ml of hot dilute hydrochloric acid and carefully swirl the mixture clockwise and anticlockwise intermittently for 3 min to 4 min. Allow to settle and carefully filter through a No. 2 Whatman or equivalent filter paper, maximum quantity of the supernatant solution into a 250 ml beaker. Give two

more washes each of about 30 ml hot acid and a final wash of about 30 ml hot water, decanting these near the rim of the filter paper cone. Heat the solution in the beaker to boil and precipitate the barium as barium sulphate by gradual addition of hot dilute sulphuric acid. Continue gentle boiling for, 10 min to 15 min to render the precipitate easily filterable. Stand for half an hour and filter through a previously ignited and weighed asbestos gooch crucible. Wash, dry, ignite, cool and weigh as in A-1.4.1.

### A-2.3 Calculation

$$\text{Barium carbonate, percent by mass} = \frac{84.58M_3}{M_4}$$

where

$M_3$  = mass, in g, of the residue in the crucible;  
and

$M_4$  = mass, in g, of the material taken for test.

## ANNEX B

[Table 1, Sl No. (ix)]

## DETERMINATION OF RELATIVE DENSITY

**B-0 GENERAL****B-0.1 Outline of the Methods**

Two methods are prescribed for determination of relative density. Method 2 which gives accurate results should be used in case of dispute. Method 1 is used for routine testing.

**B-1 APPARATUS AND REAGENTS**

**B-1.1 Pyknometer** — of 50 ml capacity

**B-1.2 Water-Bath** — to maintain  $25.0\text{ }^{\circ}\text{C} \pm 0.5\text{ }^{\circ}\text{C}$

**B-1.3 Manometer** — a suitable one

**B-1.4 Desiccator** — a suitable one to withstand a vacuum of one atmosphere

**B-1.5 Vacuum Pump**

**B-1.6 Thermometer** — A suitable thermometer having a range of  $0\text{ }^{\circ}\text{C}$  to  $60\text{ }^{\circ}\text{C}$  and an accuracy of  $0.1\text{ }^{\circ}\text{C}$  (see IS 4825).

**B-1.7 Weighing Bottle****B-1.8 Wetting Liquid**

A liquid of low evaporation rate and narrow boiling range. Generally, white kerosine of low evaporation and boiling range is suitable. Where kerosine is unsuitable, other wetting agents like glycerol, ethylene glycol, etc, may be used.

**B-1.9 Bottle**

A storage bottle (*h*, Fig. 1) for kerosine or other wetting liquid.

**B-1.10 Bell Jar**

A glass bell jar *b* with a two-hole rubber stopper. Into one hole of the stopper shall be fitted a separatory funnel with a well ground stop-cock (*c*, Fig. 1), extending into the pyknometer. Into the other hole of the stopper shall be fitted a glass tube with a well ground three-way stop-cock (*d*, Fig. 1) and connected with the vacuum pump (*c*, Fig. 1). The bell jar shall rest on a sheet of rubber, cemented or vulcanized to a glass or iron plate. With stop-cock *c* closed and stopcock *d* open to the pump, the system shall maintain an absolute pressure of at most 3 mm Hg. A desiccator may be used instead of a bell jar.

**B-2 PROCEDURE****B-2.1 Standardization of Pyknometer**

Fill the pyknometer with freshly boiled distilled water and maintain at  $25.0\text{ }^{\circ}\text{C} \pm 0.5\text{ }^{\circ}\text{C}$  and weigh after wiping the outside and making it dry. Empty

the pyknometer, clean and dry and reweigh. Next fill the pyknometer with kerosine at  $25.0\text{ }^{\circ}\text{C} \pm 0.5\text{ }^{\circ}\text{C}$ , wipe, dry and weigh. Calculate the relative density of kerosine as follows:

$$\text{Relative density of kerosine } (P), \text{ at } 25/25\text{ }^{\circ}\text{C} = \frac{M_1}{M_2}$$

where

$M_1$  = mass, in g, of kerosine; and

$M_2$  = mass, in g, of water.

**B-2.2 Method 1**

Weigh about 10 g of the pigment after transferring to a clean and dry pyknometer. Add enough kerosine to the pyknometer to form clean layer approximately 6 mm above the pigment. Place the pyknometer in desiccator which shall be closed and attached to the water pump until the greater part of the air is removed from the system. Complete the operation in about 5 min to 10 min. Close the system with a pinchcock and attach the desiccator to the oil pump for removal of small amounts of air given off at low pressures. Use the manometer to check the vacuum. When the absolute pressure is 3 mm Hg and constant, cut off the pump for short periods, taking care that the vacuum does not change materially due to leakage. Bubbles of air rise from the pigment very rapidly at first and decrease gradually and stop finally. When no more bubbles come, it may be assumed that occluded air has been removed and the material is wet with kerosine. Then slowly admit air to the desiccator by means of the pinchcock.

**B-2.2.1** Remove the pyknometer from the desiccator (bell jar), fill with kerosine at  $24\text{ }^{\circ}\text{C}$  to  $25\text{ }^{\circ}\text{C}$ , care being taken that sufficient quantity of kerosine is added to prevent air bubbles when the pyknometer is closed. Bring the pyknometer to  $25.0\text{ }^{\circ}\text{C} \pm 0.5\text{ }^{\circ}\text{C}$ . Carefully stopper the pyknometer, remove excess kerosine, wipe, dry and weigh.

**B-2.3 Calculate the Relative Density as Follows:**

$$\text{Relative density of the material} = \frac{M_1 \times P}{(M_1 + M_2) - M_3}$$

where

$M_1$  = mass, in g, of material taken;

$P$  = relative density of kerosene;

$M_2$  = mass, of the bottle in g, when filled with kerosene; and

$M_3$  = final mass of bottle in g, when filled with kerosine and pigment.



**B-2.4 Method 2**

The apparatus shall consist as shown in Fig. 1. Place the pyknometer containing the weighed, dried pigment under the bell jar. Close the stop-cocks *c* and *d* of the bell jar, start the vacuum pump, and then gradually open stop-cock *d* to the pump. When an absolute pressure of 3 mm Hg has been attained and

can be maintained, fill the separating funnel with kerosine, close stop-cock *d* and gradually open stop-cock *c*, adding sufficient kerosine to cover the pigment. Then stop the pump and release the suction at stop-cock *d*. Finally, fill pyknometer with kerosine, and complete the test as prescribed in B-2.2.1 and B-2.3.

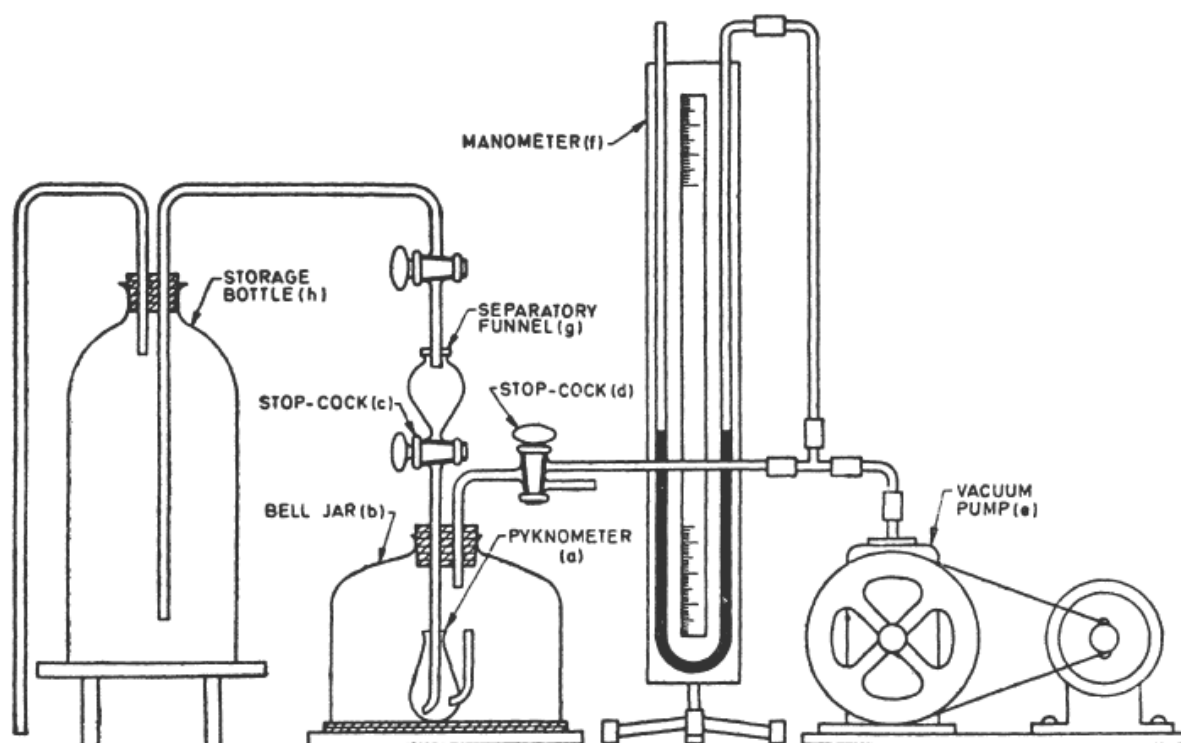


FIG. 1 APPARATUS FOR DETERMINATION OF RELATIVE DENSITY

## ANNEX C

(Foreword)

## COMMITTEE COMPOSITION

Raw Materials for Paints, Varnishes and Related Products Sectional Committee, CHD 21

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### Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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